

C L A I M S

[1] A film formation method for forming a metal  
nitride film having a predetermined thickness on a  
target substrate by heating the target substrate at a  
film formation temperature within a process container  
5 and performing a cycle comprising a first step and a  
second step at least once, such that the first step is  
arranged to supply a metal compound gas and a nitrogen-  
containing reducing gas to form a film of a metal  
10 nitride by CVD, and the second step is arranged to stop  
the metal compound gas and supply the nitrogen-  
containing reducing gas,

wherein, in film formation, the target substrate  
is set at a temperature of less than 450°C, the process  
15 container is set to have therein a total pressure of  
more than 100 Pa, and the nitrogen-containing reducing  
gas is set to have a partial pressure of 30 Pa or less  
within the process container in the first step.

[2] The film formation method according to claim 1,  
20 wherein a film thickness obtained by the cycle  
performed once is set to be 0.50 nm or less.

[3] The film formation method according to claim 1,  
wherein, in the first step, the nitrogen-containing  
reducing gas is set to have a partial pressure of 20 Pa  
25 or less within the process container.

[4] The film formation method according to claim 3,  
wherein a film thickness obtained by the cycle

performed once is set to be 2.0 nm or less.

[5] The film formation method according to claim 1, wherein, in the first step, the nitrogen-containing reducing gas is set to have a partial pressure of 15 Pa or less within the process container.

[6] The film formation method according to claim 1, wherein, in film formation, the target substrate is set at a temperature of 400°C or less.

[7] A film formation method for forming a TiN film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a Ti compound gas and a nitrogen-containing reducing gas to form a film of TiN by CVD, and the second step is arranged to stop the Ti compound gas and supply the nitrogen-containing reducing gas,

wherein, in film formation, the target substrate is set at a temperature of less than 450°C, the process container is set to have therein a total pressure of more than 100 Pa, and the nitrogen-containing reducing gas is set to have a partial pressure of 30 Pa or less within the process container in the first step.

[8] The film formation method according to claim 7, wherein the Ti compound gas is  $\text{TiCl}_4$  and the nitrogen-containing reducing gas is  $\text{NH}_3$ .

[9] The film formation method according to claim 7, wherein a film thickness obtained by the cycle performed once is set to be 0.50 nm or less.

5 [10] The film formation method according to claim 7, wherein, in the first step, the nitrogen-containing reducing gas is set to have a partial pressure of 20 Pa or less within the process container.

10 [11] The film formation method according to claim 10, wherein a film thickness obtained by the cycle performed once is set to be 2.0 nm or less.

[12] The film formation method according to claim 7, wherein, in the first step, the nitrogen-containing reducing gas is set to have a partial pressure of 15 Pa or less within the process container.

15 [13] The film formation method according to claim 7, wherein, in film formation, the target substrate is set at a temperature of 400°C or less.

20 [14] The film formation method according to claim 7, wherein, in the first step, the nitrogen-containing reducing gas is set at a flow rate of 20 mL/min or more.

[15] The film formation method according to claim 7, wherein, in the first step, the Ti compound gas is set to have a partial pressure of more than 10 Pa and not more than 50 Pa.

25 [16] The film formation method according to claim 7, wherein the TiN film is set to have a resistivity of 800  $\mu\Omega$ -cm or less.

[17] A film formation method for forming an initial metal nitride film having a first thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas; and then forming thereon an additional metal nitride film having a second thickness by continuous CVD arranged to supply a metal compound gas and a nitrogen-containing reducing gas onto the target substrate,

wherein, in formation of the initial metal nitride film, the target substrate is set at a temperature of less than  $450^{\circ}\text{C}$ , the process container is set to have therein a total pressure of more than 100 Pa, and the nitrogen-containing reducing gas is set to have a partial pressure of 30 Pa or less within the process container in the first step.

[18] The film formation method according to claim 17, wherein the first thickness is smaller than the second thickness.

[19] The film formation method according to claim 17, wherein formation of the additional metal nitride

film is performed at a film formation temperature of 450°C or more.

[20] The film formation method according to claim 17, wherein the first thickness is set to be 5 to 50 nm and the second thickness is set to be 5 to 95 nm.

[21] A film formation method for forming an initial metal nitride film having a first thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas; and then forming thereon an additional metal nitride film having a second thickness by performing a cycle comprising the first step and the second step at least once,

wherein, in formation of the initial metal nitride film, the target substrate is set at a temperature of less than 450°C, the process container is set to have therein a total pressure of more than 100 Pa, and the nitrogen-containing reducing gas is set to have a partial pressure of 30 Pa or less within the process container in the first step, and wherein, in formation of the additional metal nitride film, the nitrogen-

containing reducing gas is set to have a partial pressure of more than 30 Pa within the process container in the first step.

5 [22] The film formation method according to claim 21, wherein formation of the additional metal nitride film is performed at a film formation temperature of 450°C or more.

10 [23] The film formation method according to claim 21, wherein the first thickness is set to be 5 to 50 nm and the second thickness is set to be 5 to 95 nm.

15 [24] A film formation method for forming a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop  
20 the metal compound gas and supply the nitrogen-containing reducing gas,

wherein, in film formation, the target substrate is set at a temperature of less than 450°C, and the process container is set to have therein a total  
25 pressure of more than 100 Pa, and wherein the metal nitride film is set to have a resistivity  $R$  of  $800 \mu\Omega\text{-cm}$  or less calculated by a following formula (A);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) + 418.8 \dots (A)$$

where  $P_N$  (Pa) denotes a partial pressure of the nitrogen-containing reducing gas within the process container in the first step, and  $T_{hk}$  (nm) denotes a  
5 film thickness obtained by the cycle performed once.

[25] The film formation method according to claim 24, wherein the metal compound gas is a Ti compound gas and the metal nitride film is a TiN film.

10 [26] The film formation method according to claim 25, wherein the Ti compound gas is  $TiCl_4$  and the nitrogen-containing reducing gas is  $NH_3$ .

[27] A film formation method for forming a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a  
15 film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal  
20 nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas,

wherein, in film formation, the target substrate is set at a temperature of less than  $450^\circ C$ , and the  
25 process container is set to have therein a total pressure of more than 100 Pa, and wherein the metal nitride film is set to have a resistivity  $R$  of  $800 \mu\Omega$ -

cm or less calculated by a following formula (B);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) - 57.685 \times \ln(F_N) + 614 \dots (B)$$

where  $P_N$  (Pa) denotes a partial pressure of the nitrogen-containing reducing gas within the process container in the first step,  $T_{hk}$  (nm) denotes a film formation thickness obtained by the cycle performed once, and  $F_N$  (mL/min) denotes a flow rate of the nitrogen-containing reducing gas in the first step.

[28] The film formation method according to claim 27, wherein the metal compound gas is a Ti compound gas and the metal nitride film is a TiN film.

[29] The film formation method according to claim 28, wherein the Ti compound gas is  $TiCl_4$  and the nitrogen-containing reducing gas is  $NH_3$ .

[30] A film formation method for forming a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas,

wherein, in film formation, the target substrate



is set at a temperature of less than 450°C, and the process container is set to have therein a total pressure of more than 100 Pa, and wherein the metal nitride film is set to have a resistivity R of 800  $\mu\Omega$ -  
5 cm or less calculated by a following formula (C);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) - 57.685 \times \ln(F_N) - 2844.6 \ln(T_W) + 17658.3 \dots (C)$$

where  $P_N$  (Pa) denotes a partial pressure of the nitrogen-containing reducing gas within the process  
10 container in the first step,  $T_{hk}$  (nm) denotes a film formation thickness obtained by the cycle performed once,  $F_N$  (mL/min) denotes a flow rate of the nitrogen-containing reducing gas in the first step, and  $T_W$  (°C) denotes temperature of the target substrate.

15 [31] The film formation method according to claim 30, wherein the metal compound gas is a Ti compound gas and the metal nitride film is a TiN film.

[32] The film formation method according to claim 31, wherein the Ti compound gas is  $TiCl_4$  and the  
20 nitrogen-containing reducing gas is  $NH_3$ .

[33] A computer readable medium containing software for a computer to control a film formation apparatus, so as to form a metal nitride film having a predetermined thickness on a target substrate by  
25 heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at

least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas, wherein, in film formation, the target substrate is set at a temperature of less than 450°C, the process container is set to have therein a total pressure of more than 100 Pa, and the nitrogen-containing reducing gas is set to have a partial pressure of 30 Pa or less within the process container in the first step.

[34] A computer readable medium containing software for a computer to control a film formation apparatus, so as to form a TiN film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a Ti compound gas and a nitrogen-containing reducing gas to form a film of TiN by CVD, and the second step is arranged to stop the Ti compound gas and supply the nitrogen-containing reducing gas, wherein, in film formation, the target substrate is set at a temperature of less than 450°C, the process container is set to have therein a total pressure of more than 100

Pa, and the nitrogen-containing reducing gas is set to have a partial pressure of 30 Pa or less within the process container in the first step.

[35] A computer readable medium containing  
5 software for a computer to control a film formation apparatus, so as to form a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a  
10 cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal  
15 compound gas and supply the nitrogen-containing reducing gas, wherein, in film formation, the target substrate is set at a temperature of less than 450°C, and the process container is set to have therein a total pressure of more than 100 Pa, and wherein the  
20 metal nitride film is set to have a resistivity R of 800  $\mu\Omega$ -cm or less calculated by a following formula (A);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) + 418.8 \dots (A)$$

where  $P_N$  (Pa) denotes a partial pressure of the  
25 nitrogen-containing reducing gas within the process container in the first step, and  $T_{hk}$  (nm) denotes a film thickness obtained by the cycle performed once.

[36] The medium according to claim 35, wherein the metal compound gas is a Ti compound gas and the metal nitride film is a TiN film.

[37] A computer readable medium containing  
5 software for a computer to control a film formation apparatus, so as to form a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a  
10 cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal  
15 compound gas and supply the nitrogen-containing reducing gas, wherein, in film formation, the target substrate is set at a temperature of less than 450°C, and the process container is set to have therein a total pressure of more than 100 Pa, and wherein the  
20 metal nitride film is set to have a resistivity R of 800  $\mu\Omega$ -cm or less calculated by a following formula (B);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) - 57.685 \times \ln(F_N) + 614 \dots (B)$$

25 where  $P_N$  (Pa) denotes a partial pressure of the nitrogen-containing reducing gas within the process container in the first step,  $T_{hk}$  (nm) denotes a film

formation thickness obtained by the cycle performed once, and  $F_N$  (mL/min) denotes a flow rate of the nitrogen-containing reducing gas in the first step.

[38] The medium according to claim 37, wherein the metal compound gas is a Ti compound gas and the metal nitride film is a TiN film.

[39] A computer readable medium containing software for a computer to control a film formation apparatus, so as to form a metal nitride film having a predetermined thickness on a target substrate by heating the target substrate at a film formation temperature within a process container and performing a cycle comprising a first step and a second step at least once, such that the first step is arranged to supply a metal compound gas and a nitrogen-containing reducing gas to form a film of a metal nitride by CVD, and the second step is arranged to stop the metal compound gas and supply the nitrogen-containing reducing gas, wherein, in film formation, the target substrate is set at a temperature of less than  $450^{\circ}\text{C}$ , and the process container is set to have therein a total pressure of more than 100 Pa, and wherein the metal nitride film is set to have a resistivity  $R$  of  $800\ \mu\Omega\text{-cm}$  or less calculated by a following formula (C);

$$R = 115.75 \times \ln(T_{hk}) + 71.576 \times \ln(P_N) - 57.685 \times \ln(F_N) - 2844.6 \ln(T_W) + 17658.3 \dots (C)$$

where  $P_N$  (Pa) denotes a partial pressure of the  
nitrogen-containing reducing gas within the process  
container in the first step,  $T_{hk}$  (nm) denotes a film  
formation thickness obtained by the cycle performed  
5 once,  $F_N$  (mL/min) denotes a flow rate of the nitrogen-  
containing reducing gas in the first step, and  $T_W$  ( $^{\circ}\text{C}$ )  
denotes temperature of the target substrate.

[40] The medium according to claim 39, wherein the  
metal compound gas is a Ti compound gas and the metal  
10 nitride film is a TiN film.